AD-A250 088

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Report No. NADC-91093-60
Contract No. N62269-90-M-7269

SURFACE ANALYSIS OF STAINLESS STEEL OUTER RACE BEARING SPECIMENS

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OCTOBER 1991

FINAL REPORT

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Prepared for
Air Vehicle and Crew System Technology Department (Code 6062)
NAVAL AIR DEVELOPMENT CENTER
Warminster, PA 18974-5000

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REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (*Leave blank*)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	October 1991	Final

4. TITLE AND SUBTITLE

Surface Analysis of Stainless Steel Outer Race Bearing Specimens

5. FUNDING NUMBERS

C: N62269-90-M-7269

6. AUTHOR(S)

D.K. Schaffer and H.M. Hand

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

MARTIN MARIETTA CORPORATION
Martin Marietta Laboratories
1450 South Rolling Road
Baltimore, MD 21229

8. PERFORMING ORGANIZATION

MML TR 91-16c

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

Air Vehicle and Crew Systems Technology
Department (Code 6062)
NAVAL AIR DEVELOPMENT CENTER
Warminster PA 19874-5000

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

NADC-91093-60

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION AVAILABILITY STATEMENT

Approved for Public Release;
Distribution is Unlimited

12b. DISTRIBUTION CODE

13. ABSTRACT (*Maximum 200 words*)

Executive Summary

The reaults of analysis of R-4 stainless steel instrument bearings, subjected to various wear cycles, are reported to describe the fate of a synthetic Schiff base lubricant additive at the bearing wear track surfaces. The surfaces were monitored by x-ray, photoelectron spectroscopy (XPS), and Fourier infrared (FTIR) spectroscopy to characterize all lubricant (and grease) species of interest.

The data indicate a general modification or degradation of both the lubricant and ubiquitous fluorinated grease ("Krytox') additive during the wear stages. Chemical and structural compositions are described for residual derivatives at the bearing track surfaces. Further studies are recommended that will enable Confirmation of both the chemical fate and the molecular mechanism of lubricant additives candidates.

14. SUBJECT TERMS	15. NUMBER OF PAGES
R-4 stainless steel bearings, Schiff base lubricant additive, Fourier infared (FTIR), x-ray, photoelectron spectroscopy (XPS)	26
	16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	Unlimited

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INTRODUCTION

Wear of manufactured products, such as stainless steel instrument bearings, is a natural dissipative process which eventually leads to inadequate performance. Tribological dissipative processes, related to the motion between two surfaces in contact, can effect conditions at surface interfaces which cause component or system failure.

Worn parts typically result in increased vibration and fatigue, shock loading and misalignment of equipment, and wear debris can cause seizure or spalling faliures in other components. Even if failure does not occur, the deterioration in performance caused by wear leads to significant losses in energy and efficiency of mechanical systems.

The use of lubricants to resist wear provides several functions: friction reduction, cooling, surface separation by fluid film generation and wear reduction. Lubricated rolling wear, as experienced by bearings at wear track surfaces, can be reduced by choosing lubricants with optimal compatibility (i.e. viscosity, cooling capacity, structural design, etc.) with the particular system and service conditions (temperature, load, duty cycle, environment) required.

BACKGROUND

An initial lot of four specimens, cut from outer race sections of R-4 stainless steel 440°C instrument bearings, was submitted for study of the fate of a [salicylaldehyde/benzidine] Schiff base additive (4,4'-Benz, or "Benz") at the bearing wear track surface after wear cycling. The specimens were designated as follows:

PT	Pretreated by refluxing with 4,4'-Benz Schiff base; no outer race wear track (not run in bearing test)
358	Run in bearing test to failure (71 hr) with the grease only
94B	Run in bearing test with grease containing 5% Schiff base additive; removed prior to failure (400+ hr)
N23	Run in bearing test with grease containing 5% Schiff base additive; removed prior to failure (400+ hr)

Following analysis of these four initial specimens [MML Interim Report, 8/15/90], three additional specimens were submitted for surface characterization. The three new specimens were designated as follows:

385	4,4'-Benz baseline (+ grease): not run to failure (40 hr)
380	4,4'-Benz (+ grease): not run to failure (400 hr)
95B	4,4'-Benz (+ grease): run to failure (728 hr)

The raceway specimens were examined by x-ray photoelectron (XPS) and Fourier transform infrared (FTIR) spectrophotometric methods to determine the chemical composition of their respective surfaces. A bearing race specimen, showing the respective wear (on track) and non-wear (off track) regions analyzed, is illustrated in Fig. 1.

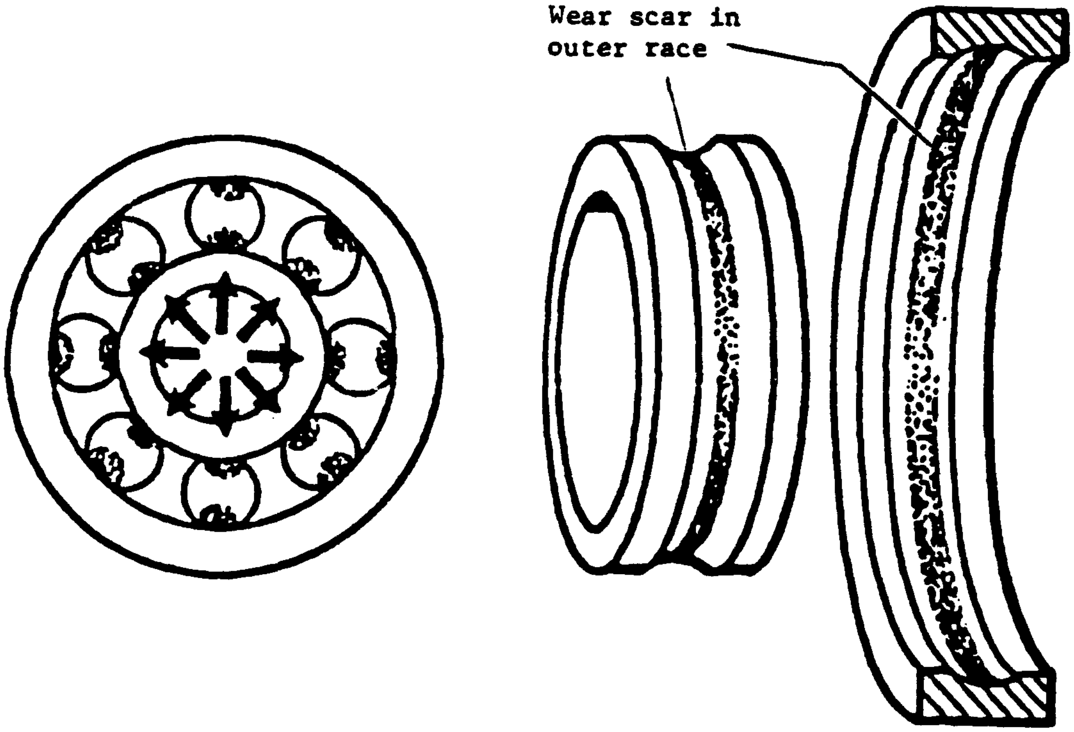


Figure 1. Ball path indicating excessive shaft mounting fit.

XPS ANALYSIS

Analysis of the surface composition (top ~2 nm) was achieved using x-ray photoelectron spectroscopy (XPS). For most specimens, survey spectra were obtained for areas both on and off the wear tracks. Analysis was performed using a Surface Science Instruments SSX 100-03 spectrometer with a monochromatized Al x-ray source focussed to a 300 um spot. Quantification was acheived using the peak areas of the primary photoemission peak for each element and sensitivity factors provided by the manufacturer and/or calculated from standards.

The XPS results for all of the raceway specimens, including a "clean" control sample, are presented in Table 1. The control, PT and 358 specimens were analyzed in areas both on and off of the wear track surface, while 94B and N23 were analyzed only in the wear track region. The nitrogen (N) and fluorine (F) signals served as "fingerprints" for the Benz and grease, respectively. Similarly, the iron (Fe), aluminum (Al) and chromium (Cr) represent the bearing material.

The "clean" (control) bearing specimen indicated primarily carbon (C) and oxygen (O), with lesser quantities of Fe, Al and Cr, corresponding to an oxidized metal surface covered with nominal adventitious hydrocarbons. The higher C and lower O concentrations on the wear track surface suggest a comparatively thicker organic (C) layer on top of a less oxidized track surface. The change in the relative concentrations of the metals may indicate wear which served to expose material from below the surface of the bearing.

The Benz-pretreated (PT) and run-to-failure (358) bearing surfaces were comparable in composition, with a C/O ratio of ~2.5, rangng from 1.7 (358, off track) to 2.8

TABLE 1
XPS RESULTS FOR RACEWAY SPECIMENS

Specimen	C	O	Fe	Al	Cr	Si	S	F	N	Ca	K	P	Na	Cl
Control (off track)	47.28	35.74	8.06	5.18	3.74	-----	-----	-----	-----	-----	-----	-----	-----	-----
Control (on track)	62.74	22.98	2.42	6.05	5.68	-----	-----	-----	-----	-----	-----	-----	-----	-----
PT (off track)	65.23	22.99	1.48	4.92	2.02	2.44	0.92	-----	-----	-----	-----	-----	-----	-----
PT (on track)	62.52	26.11	1.87	5.85	1.78	1.87	-----	-----	-----	-----	-----	-----	-----	-----
358 (off track)	54.42	32.77	1.56	2.46	1.67	6.31	0.81	-----	-----	-----	-----	-----	-----	-----
358 (on track)	59.01	29.86	1.56	2.28	1.50	4.81	0.98	-----	-----	-----	-----	-----	-----	-----
94B (on track)	28.04	10.84	-----	-----	-----	0.81	-----	48.84	3.57	0.80	5.64	1.46	-----	-----
N23 (on track)	12.73	17.94	2.25	-----	2.28	-----	0.63	46.59	2.79	-----	4.92	2.06	7.79	-----
385 (off track)	48.08	12.34	-----	-----	-----	-----	-----	32.42	7.16	-----	-----	-----	-----	-----
385 (on track)	59.94	18.34	-----	-----	-----	1.45	-----	9.[illegible]5	10.71	-----	-----	0.12	-----	-----
380 (off track)	43.06	6.94	-----	-----	-----	-----	-----	48.97	1.05	-----	-----	-----	-----	-----
380 (on track)	38.12	4.21	-----	-----	-----	-----	-----	50.26	1.18	-----	-----	-----	-----	0.23
95B (off track)	45.20	9.25	0.55	-----	0.44	-----	-----	41.64	2.92	-----	-----	-----	-----	-----
95B (on track)	53.81	18.32	1.05	-----	-----	1.41	-----	20.00	5.41	-----	-----	-----	-----	-----

(PT, off track). In addition to Fe, Al and Cr, varying quantities of silicon (Si, 1.9-6.3%) and sulfur (S, 0.8-1.0%) were also detected on these surfaces.

The absence of the Schiff base additive on the grease from the PT and 358 surfaces (indicated by the absence of N and F, respectively) was somewhat surprising. It is possible, although not confirmed, that the fluroinated grease [MIL-G-10924E/Dupont Krytox GPL] was combusted during the test carried to failure (358).

The Benz and grease are clearly present on the 94B and N23 bearing surfaces. A thicker grease film, especially on 94B, is indicated by the attenuation of the metal signals. In addition, both specimens indicated varying quantities of potassium (K), phosphorus (P), calcium (Ca) and/or sodium (Na).

As described earlier, the F was attributed specifically to the fluorinated grease and accounted for nearly half of the composition of the residual surface layers for the 94B and N23 bearing specimens. Similarly, N (2.8-3.6%) was considered as a marker for the 4,4'-Benz (-C=N-) additive. The elevated C + N levels suggest a somewhat higher residual Schiff base concentration on the 94B surface (relative to N23).

XPS analysis of the second lot of raceway specimens describes the progressive fate of the bearing surfaces during cycling. The dominant element on the surface of specimen 385 is C (60% on track, 48% off track), with a significant amount of N (10.7% on, 7.2% off) indicating the presence of the "baseline" Benz additive. The diminished F content, particularly on the wear track (9.5%), indicates a lower concentration of grease at the 385 surface. The reduced O and metal levels are attributed to a thicker grease/Benz layer relative to the control and PT (pretreated, not tested) bearing specimens.

The 380 specimen (not run to failure, 400 hr) contained comparatively less C, N and O, and significantly more F than the 385 Benz baseline wear surfaces. The 89% (on-track) reduction in surface N concentration suggests that the Benz Schiff base additive has either migrated toward or into the bearing matrix or has been combusted to a considerable extent. The corresponding decrease in surface C (by 37%, on track) and O (by 78%, on track) is consistent with the migration/combustion of the salicylaldehyde-Benz derivative. This depletion is accompanied by an increase in surface [F], the diagnostic elemental marker for the grease. Whether the change is due to a "migration" of the grease or represents only sample-to-sample variation, cannot be ascertained by these XPS results alone.

Finally, the surface of the 95B specimen (run to failure, 728 hr) indicated that the C and O concentrations have been restored to their approximate levels in the 385 specimen, and N to about half of its original concentration. These increases occurred at the expense of F, which was reduced (by 64% on track and 15% off track), relative to the intermediate (400 hr) 380 specimen. Fe also was detected, suggesting that the overall protective layer (Benz + grease) had thinned enough to expose the bearing surface itself, at least in some areas of the track.

FTIR Analysis

Fourier Transform Infrared (FTIR) Analysis was performed using a Nicolet 5DXC spectrophotometer equipped with SX software and a HgCdTe detector (for increased sensitivity). All spectra were obtained in dry, CO_2-free air. The 4,4'-Benz standard was run on a KBr pellet, and the base fluorinated grease and grease + 4,4'-Benz mixture samples were run neat on a NaCl disc. The racer bearings were analyzed using FTIR in the specular reflectance "thin coatings reflectance" mode.

During each run the raceway sample was mounted in an Accuspec Model 2000 multi-mode FTIR cell, which was aligned in the sample compartment of the spectrophotometer. Specular reflectance FTIR analysis was performed on each specimen with the beam at a 10° angle of incidence to the sample surface. Both 5000 and 20,000 scan cycles were collected for each specimen. Spectra illustrating the instrument background signature (a) and outer race control pattern (b) are included in Figure 2.

Transmission spectra of pure 4,4'-Benz (KBr) and the fluorinated grease are shown in Figures 3a and 3b, respectively. The Benz Schiff base is characterized by the -C=N- stretching bands in the 1689-1471 cm^{-1} region. Diagnostic peaks also appear in aromatic C-H stretching (3057 cm^{-1}), out-of-plane (900-700 cm^{-1}) and in-plane (1290-1125 cm^{-1}) absorption regions.

The fluorinated grease spectrum indicates strong peaks in the 1310-800 cm^{-1} region resulting from C-F stretching modes. The absence of bands in the 3000-2900 cm^{-1} (C-H stretching) and 1425 cm^{-1} (C-H deformation) regions confirm the polytetrafluoroethylene (PTFE) type of structure.

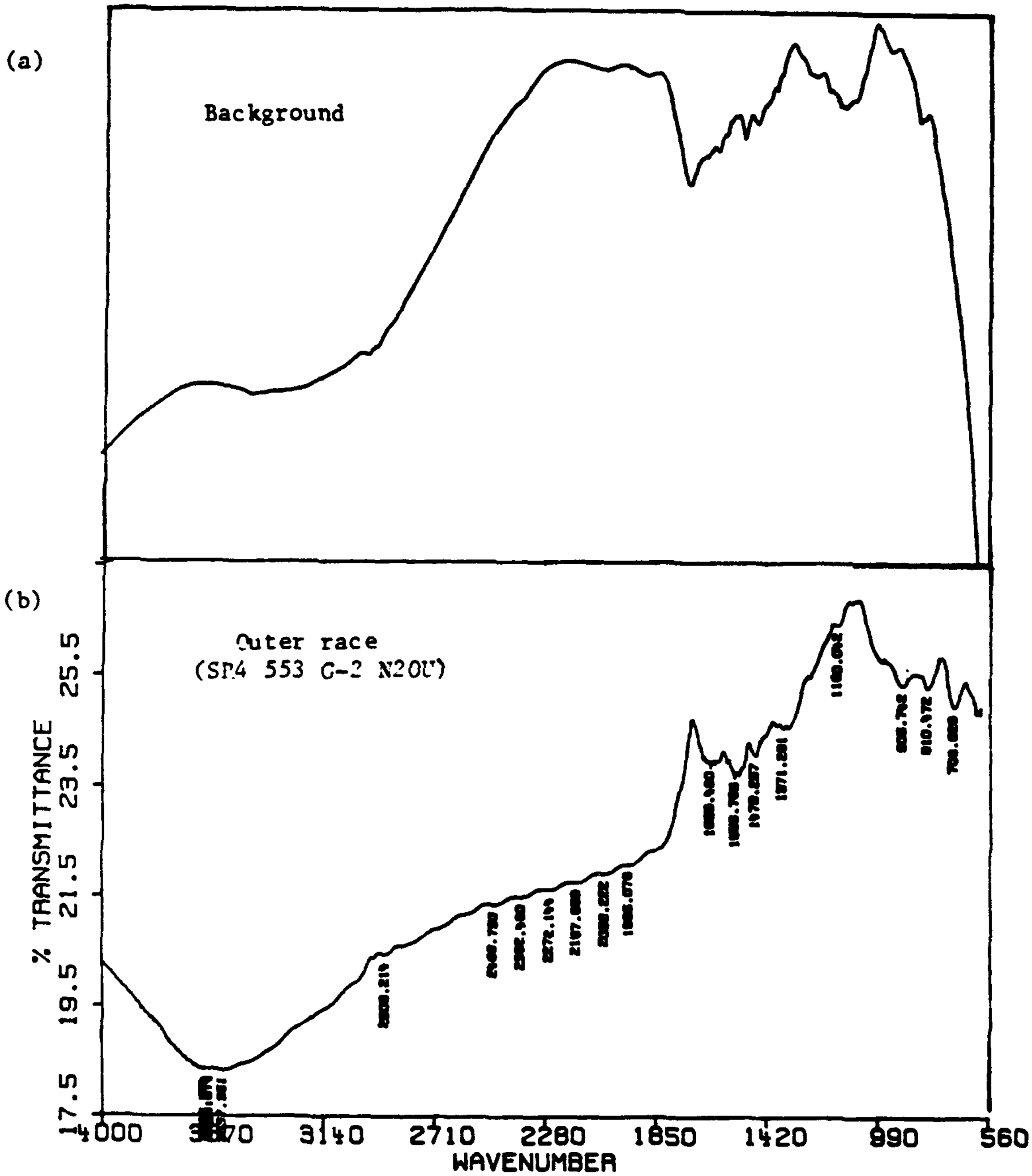


Figure 2. FTIR spectra of (a) instrument background and (b) outer race control surface.

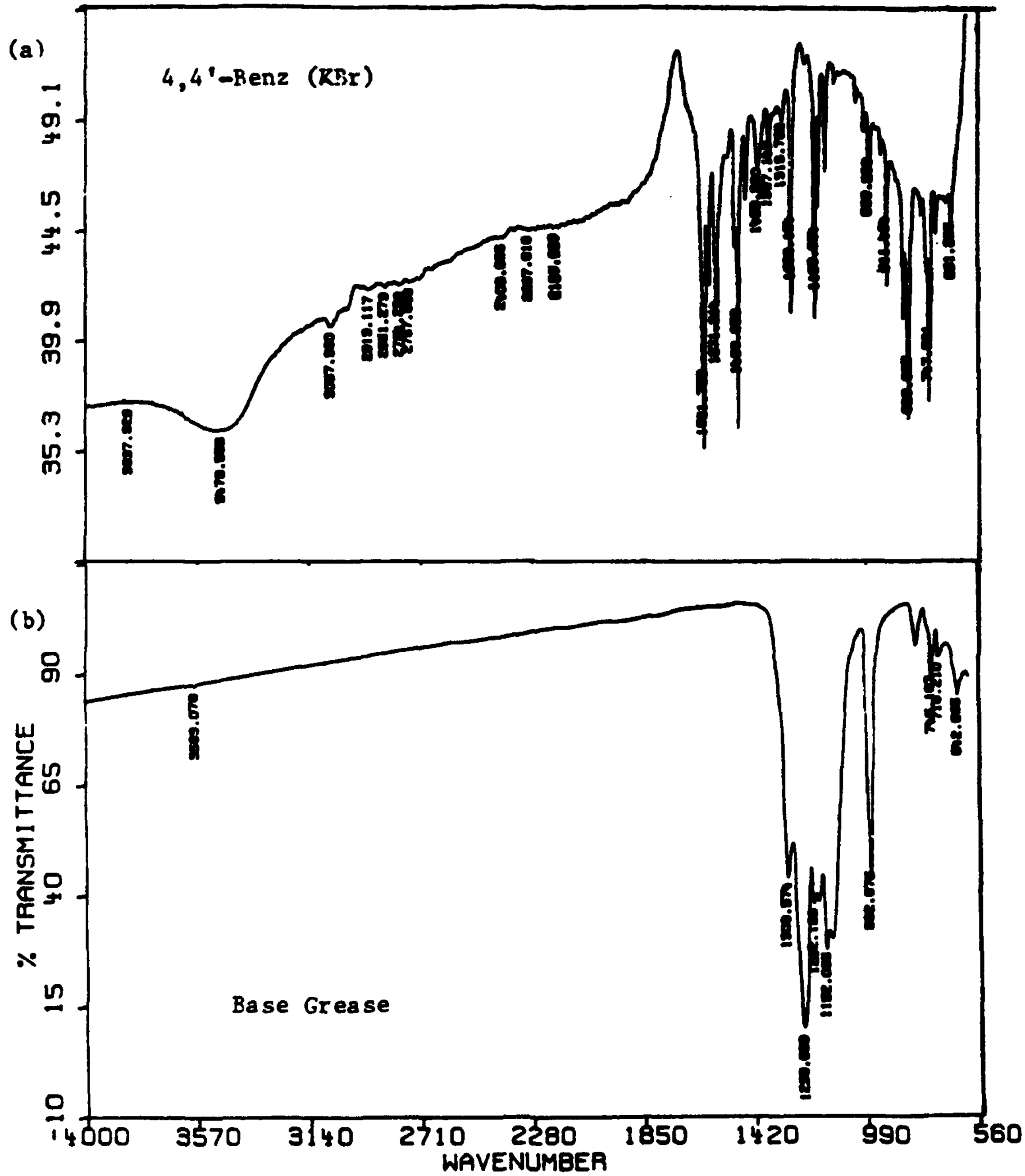


Figure 3. FTIR spectra of pure (neat) standard materials: (a) 4,4'-Benz Schiff base and (b) fluorinated grease.

Mixtures of the grease and 4,4'-Benz additive (approx. 5% w/w) yield spectra that are dominated by the grease-affiliated peaks (Fig. 4). The out-of-plane aromatic C-H peaks of the Schiff base are coincident with the C-F stretching frequencies in the grease (no utility). Thus, the -C=N-stretching bands represent the only diagnostic markers for the additive.

Extensive reflectance analysis of the uncycled (PT) and baseline failure (358) bearing surfaces failed to detect meaningful IR absorption beyond the nominal outer race background signature (Figs. 5 and 6, respectively). The possibility exists that faint diagnostic 4,4'-Benz peaks (1689-1471 cm^{-1} region) are being masked by background matrix (i.e. steel) absorption bands on the PT surface.

However, the primary C-F stretching bands (1257, 1202 and 1152 cm^{-1}) are clearly absent in the 358 specimen (in a "clean" diagnostic region), indicating that the grease has disappeared (or undergone drastic chemical modification) due to the bearing cycling process.

The 94B and N23 surfaces (Figures 7a and 7b, respectively) indicate non-background diagnostic peaks, particularly in the C-F absorption region. However, the PTFE-type profile has been significantly altered (absence of 1308, 1238, 1202 and 962 cm^{-1} peaks), indicating a chemical modification of the grease during cycling.

FTIR analysis of the 385 (40 hr, not run to failure) outer raceway surface (Figure 8a) indicated the presence of 4,4'-Benz (1622, 1479 cm^{-1}). The 385 inner raceway surface (Figure 8b) contained no diagnostic grease peaks (1400-1000 cm^{-1} range), and possible 4,4'-Benz peaks were masked by additional absorption in the 1630-1400

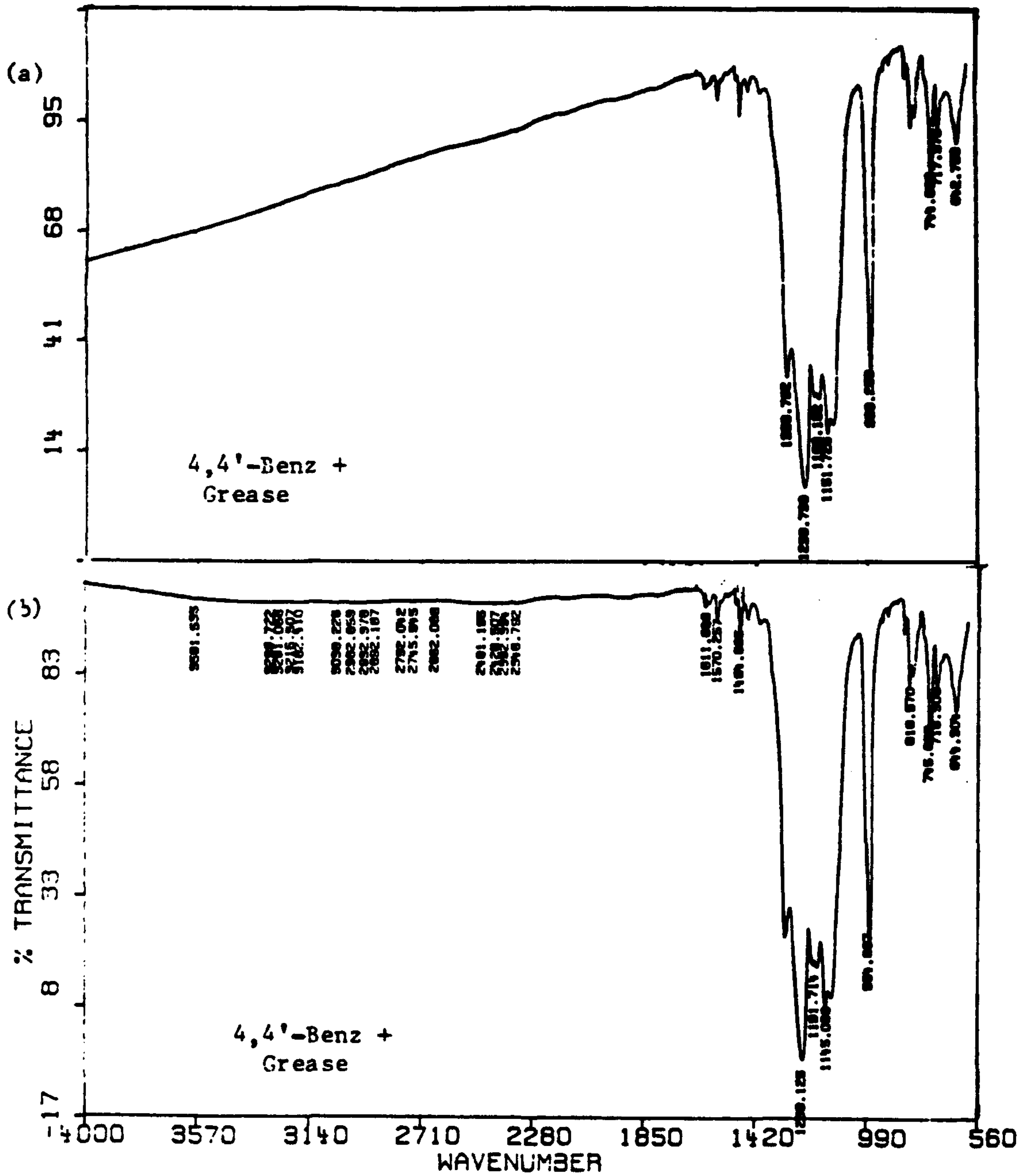


Figure 4. FTIR spectra of mixture of 4,4'-Benz + Grease collected at two different incident angles.

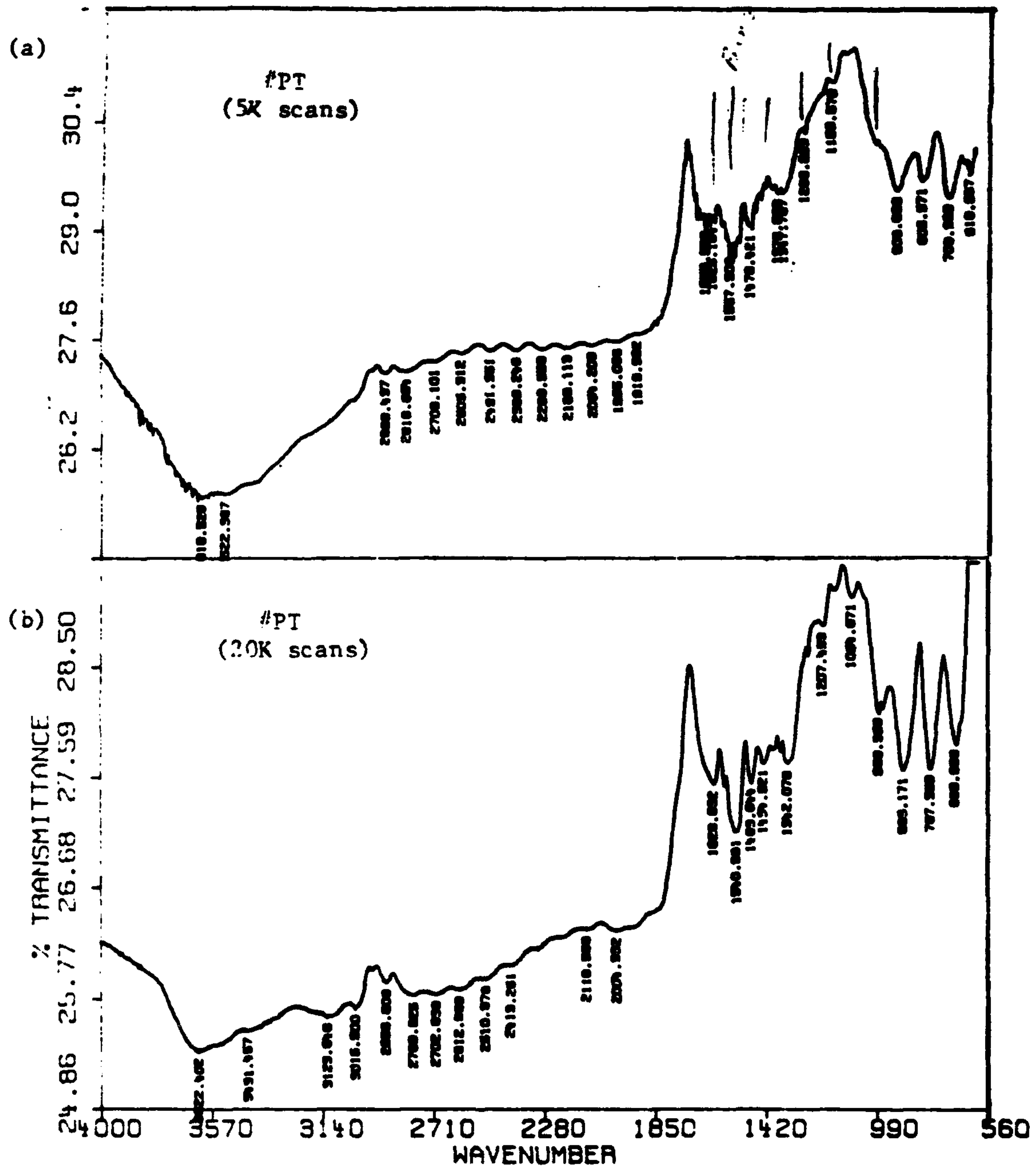


Figure 5. FTIR spectra of PT (pretreated) bearing specimen after (a) 5,000 and (b) 20,000 scans.

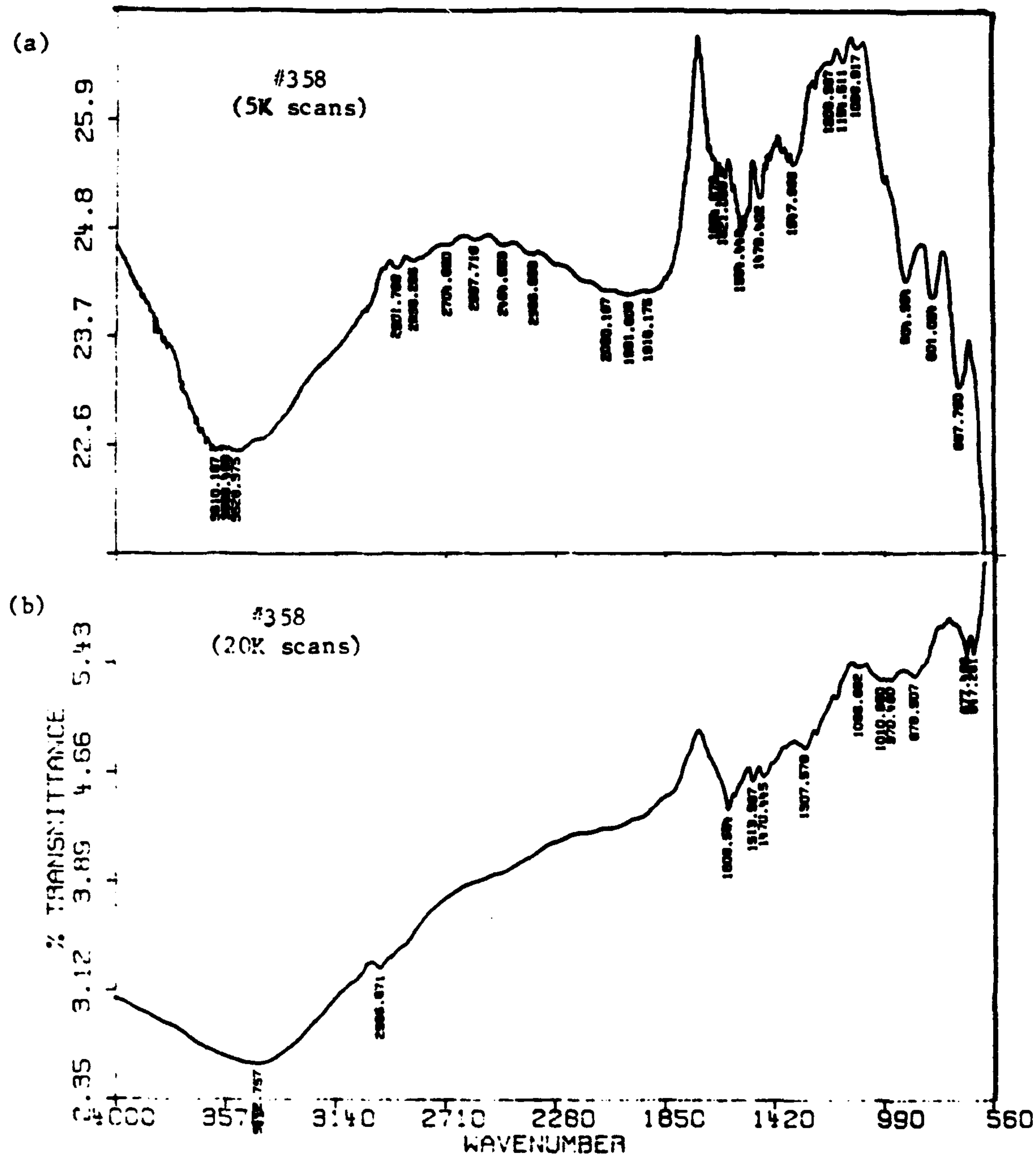


Figure 6. FTIR spectra of bearing specimen #358 (baseline) after (a) 5,000 and (b) 20,000 scans.

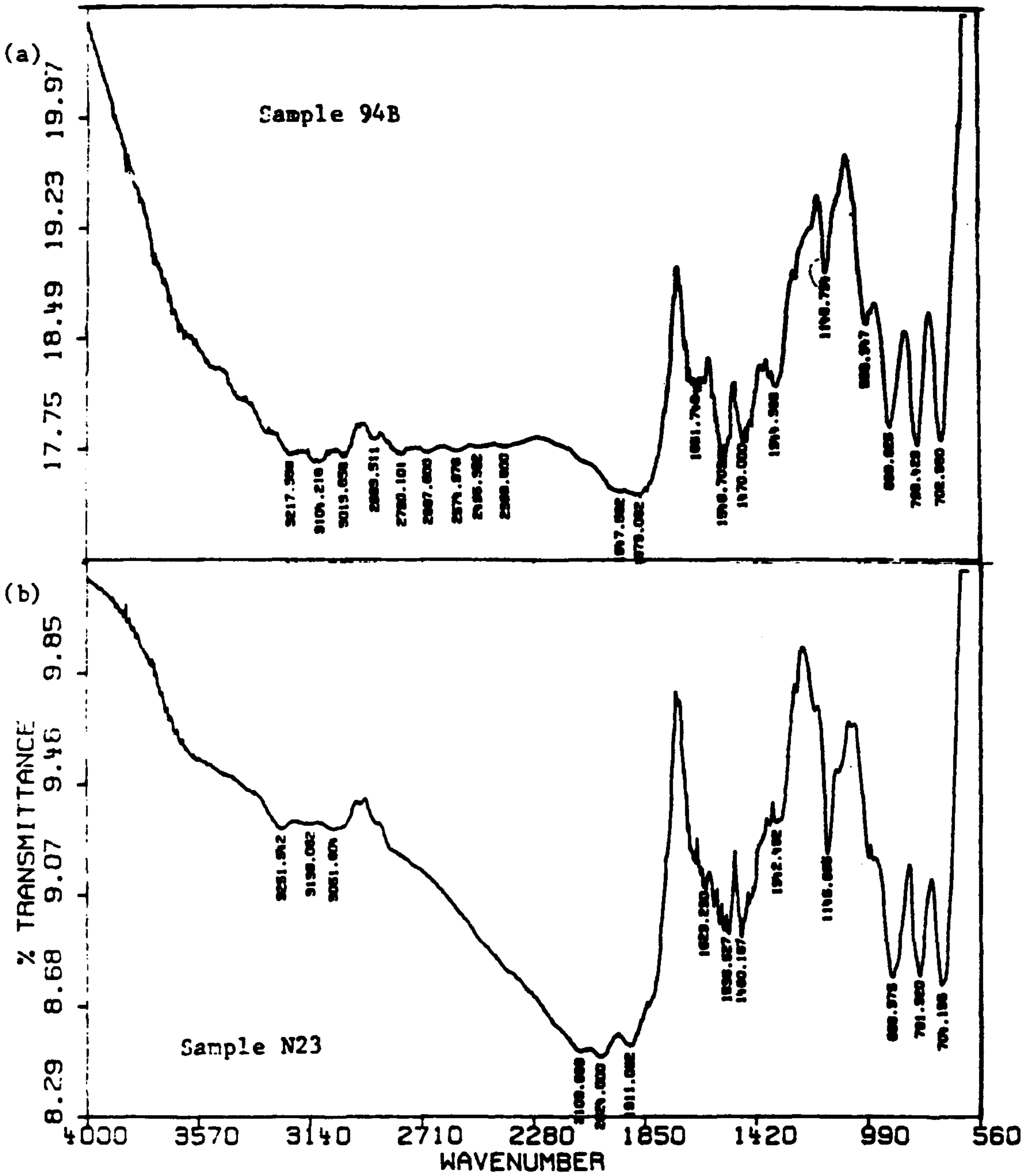
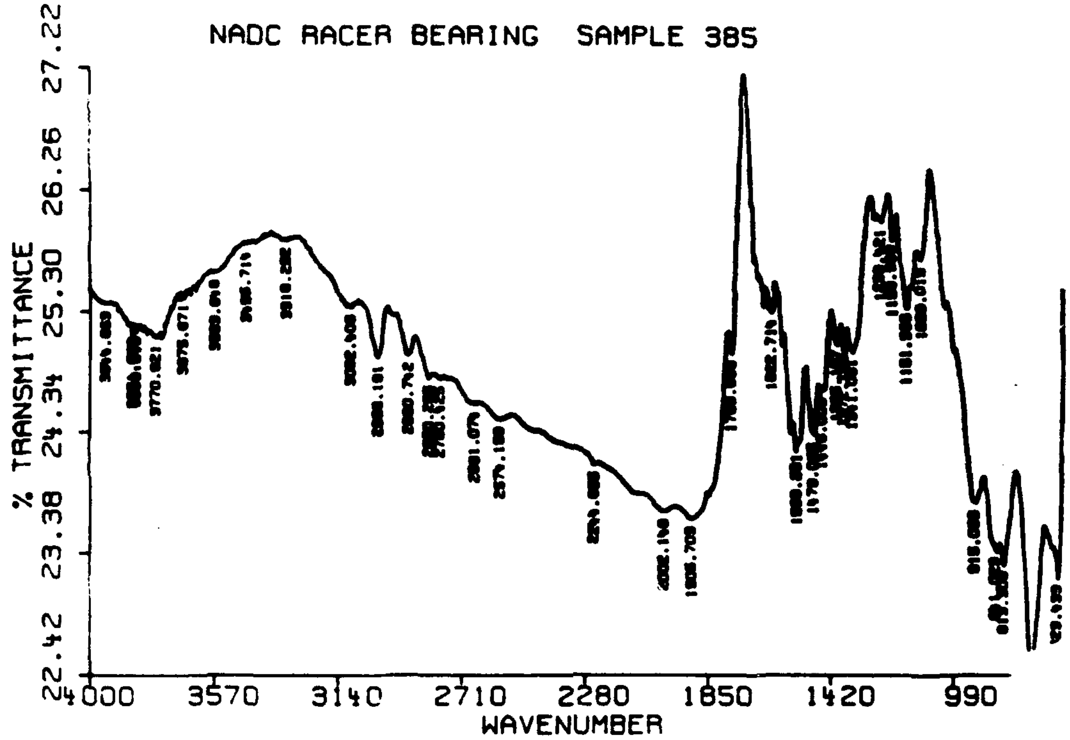


Figure 7. FTIR spectra of (a) 94B and (b) N23 bearing specimen surfaces.

(a)



(b)

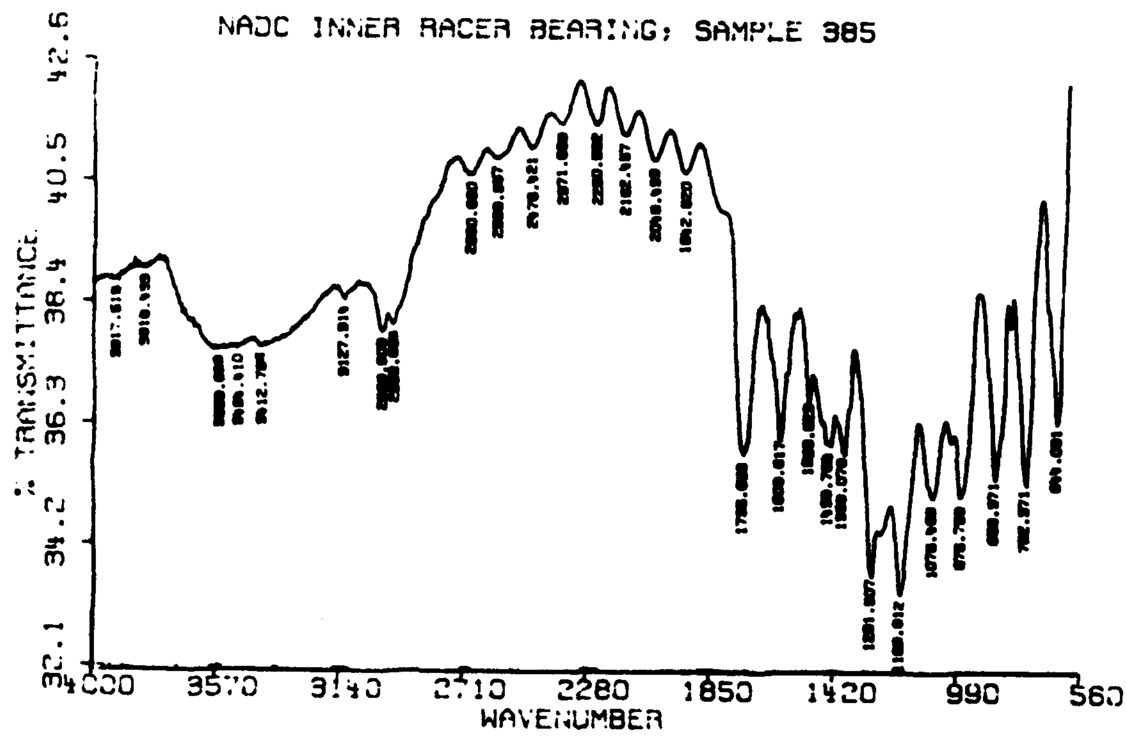


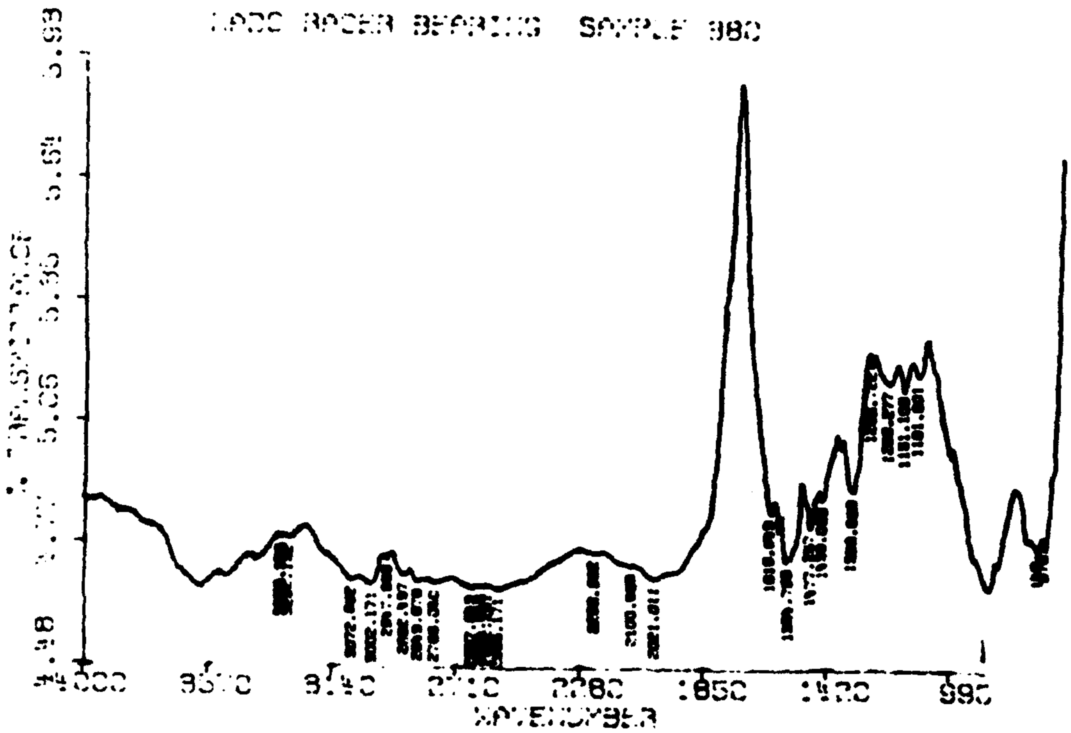
Figure 8. FTIR spectra of (a) outer and (b) inner raceway 385 bearing specimens.

cm^{-1} region, making positive identification difficult. Peaks in the aromatic (3130-3070 cm^{-1}) region support the presence of the Schiff base complex on the 385 inner surface.

The 380 (400 hr, not to failure) outer raceway (Figure 9) track indicated traces of the grease compound (1151 cm^{-1}), although not to the extent suggested by the XPS results. The inner track, however, appeared to contain an appreciable quantity of grease, as indicated by the strong, sharp peaks at 1207, 1159 and 1102 cm^{-1}. This result is consistent with the high (56%) F level detected by XPS on the 380 inner surface.

FTIR surface examination of specimen 95B (728 hr, run to failure) indicated the presence of 4,4'-Benz (1532, 1489 cm^{-1}) on the inner raceway track (Figure 10). However, lesser quantities of the Schiff base were observed on the outer surface, which is in agreement with XPS results. Further analysis revealed more of the grease component in the outer track; however, the diagnostic (1238 cm^{-1}) absorbance peak was shifted by 39 cm^{-1} (1199 cm^{-1}), indicating a different chemistry for the fluorinated grease, relative to the 380 baseline specimen. In fact, the same absorbance triad (1206, 1151, 1102 cm^{-1}) was observed for the 380 outer surface. This may indicate a degradation or rearrangement of the grease resulting from the prolonged thermal cycling treatment.

(a)



(b)

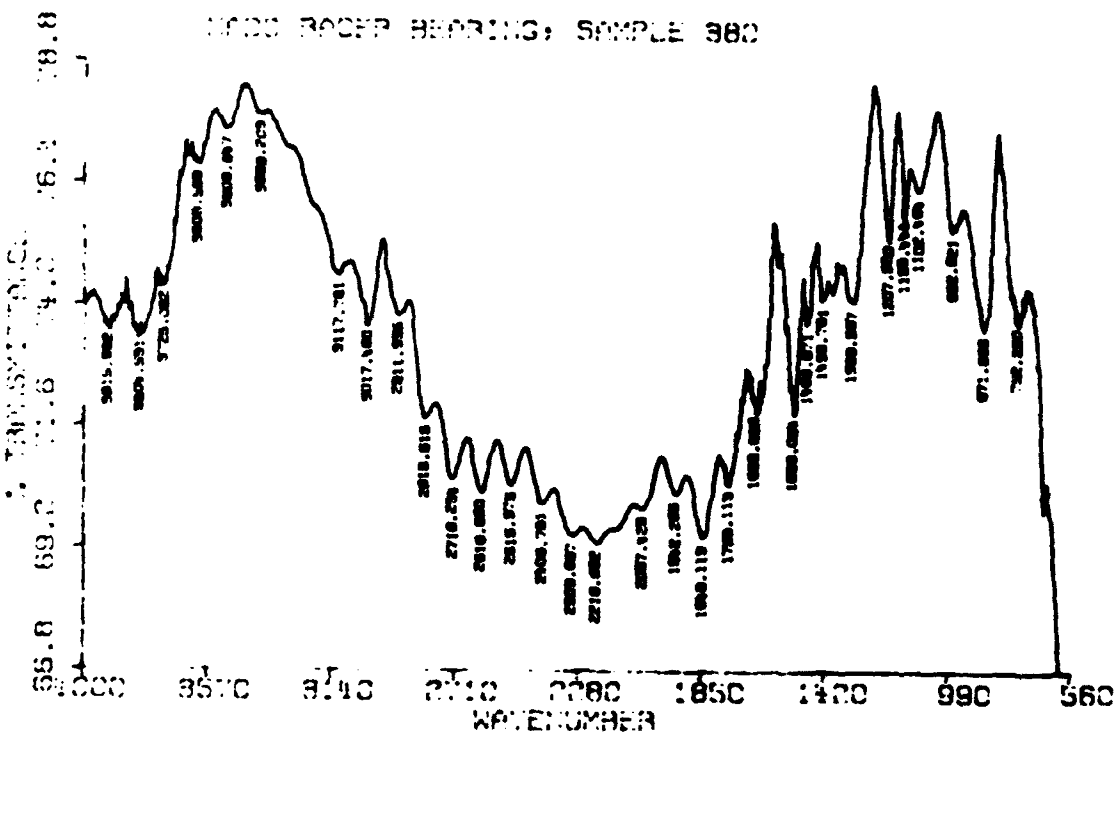
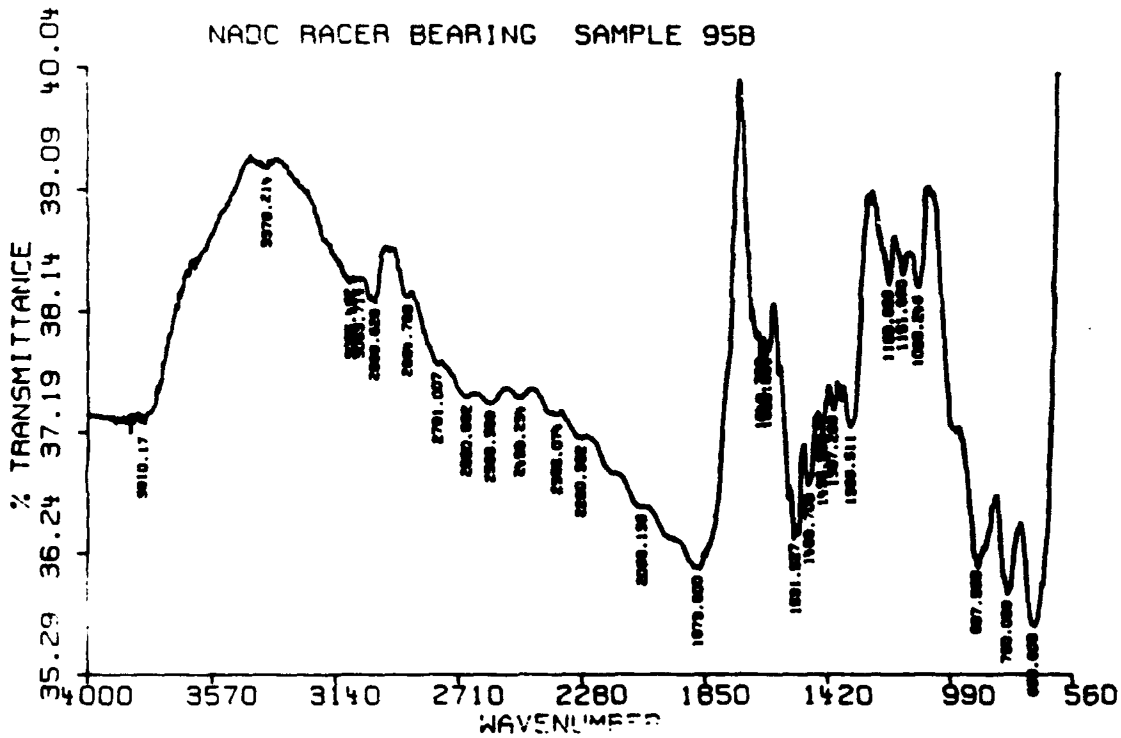


Figure 9. FTIR spectra of (a) outer and (b) inner raceway 380 bearing specimens.

(a)



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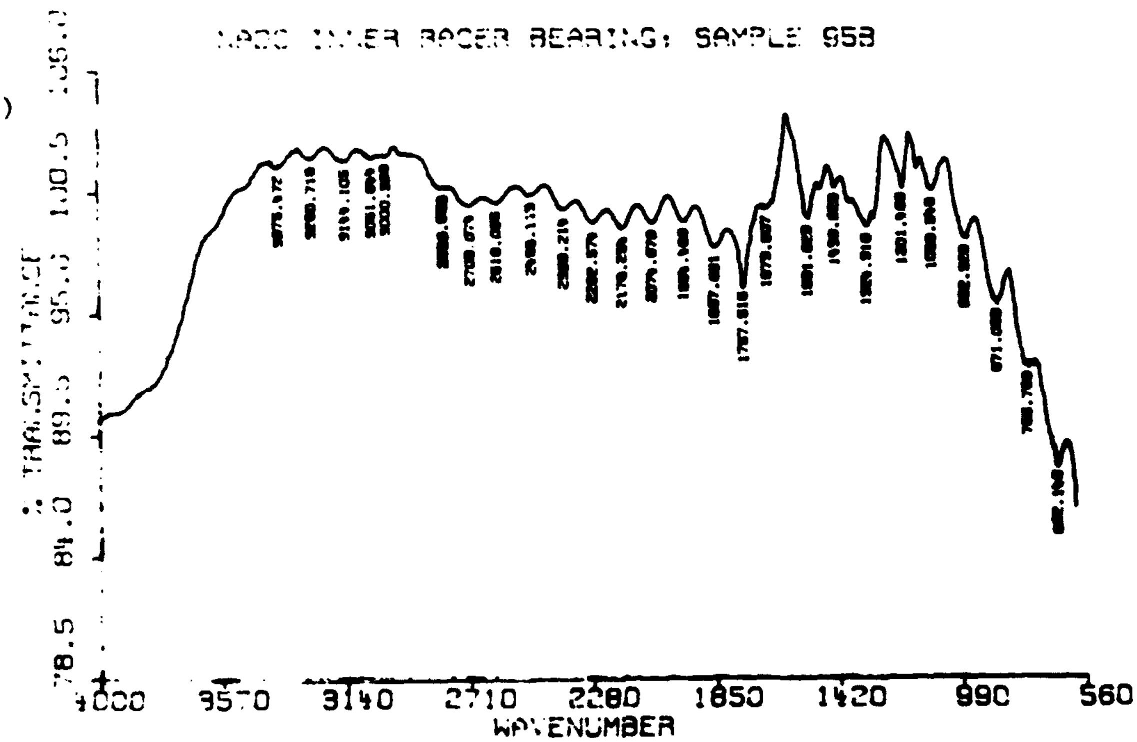


Figure 10. FTIR spectra of (a) outer and (b) inner raceway 95B bearing specimens.

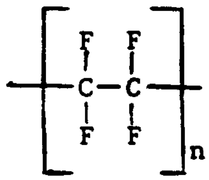
DISCUSSION AND CONCLUSIONS

The analysis of bearing surfaces that have undergone wear testing indicate that significant chemical changes occur during the cycling process. For initial specimen lots run in the bearing test with grease containing 5% Benz and removed prior to failure (94B, N23), the surface consisted of ~50% F (at. %), with 3-4% N and 12-28% C detected. An examination of the structural compositions of the grease and Benz Schiff base compounds suggests some anomolies with respect to assignments for pure reference materials (Fig. 11).

For example, if all of the F detected in 94B and N23 represents unmodified PTFE-type grease, the F/C ratio of 2.3 requires that approximately 21% of these surfaces consist of C. This is theoretically possible for 94B (28% total C), but not for N23 (13%C). Similarly, if the N on these surfaces indicates exclusively pure 4,4'-Benz lubricant, the required C concentrations (C/N ~ 11.4) amount to 41% for 94B and 32% for N23. The corresponding C levels that would be required for unmodified grease + lubricant materials, based on the F and N values determined, amount to 62% (21+41) C for 94B and 53% (21+32) for N23. Thus, it is obvious that significant chemical modifications of the grease + Benz compounds occurred during testing.

The prediction of chemical modification of the 94B and N23 grease + lubricant additives during wear cycling is supported by the FTIR results. While the detection levels are not as sensitive for surface-adsorbed (vs. neat) materials, FTIR analysis clearly indicates significantly "simplified" structures in the regions of highest diagnostic utility -- suggesting a partial degradation and/or combustion of the additive materials. Definitive qualitative peak assignments were not made, and are not recommended without performing parallel controlled testing of treated witness specimens.

(a)



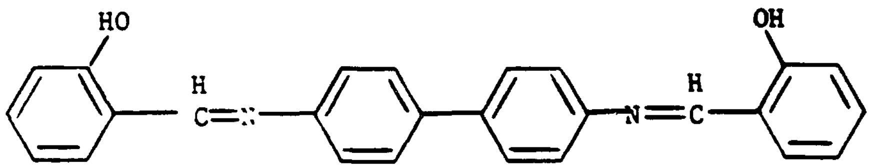
$n \geqslant 20{,}000$

Atomic composition

C = 30%
F = 70%

F/C = 2.3

(b)



Atomic composition

C = 80%
O = 8%
N = 7%
H = 5%

C/N = 11.4

Figure 11. Chemical structures of (a) fluorinated (PTFE-type) grease and (b) 4,4'-Benz Schiff base lubricant additives

Comparable surface analyses of more recent lots of test specimens proved insightful, with regard to structure determination of the [grease + lubricant] additives. For specimen 385, a [F] concentration of 32% (off track) corresponds to a theoretical C level of 14%; however, the high [N] projects to 82% C, if the Schiff base remains unmodified during cycling. This is not possible, of course, suggesting that some chemical modification occurred.

The FTIR pattern of 385 is similar to that observed for 94B and N23; the lower [F] (385 on track) corresponds to a low (4%) C level, while the higher [N] requires a theoretical [C] level of 122%. Thus, the N-rich compound on the surface does not indicate (entirely) unmodified Schiff base; in fact the FTIR spectrum is distinctly barren in the key -C=N- absorbance regions. Peaks were retained, however, in the 1609 and 1508 cm^{-1} (-C=C-) areas and additional peaks show increased intensity in the 1292 and 1183 cm^{-1} locations. A trace of aromatic -C-H- absorbance (3128 cm^{-1}) also remains. Qualitative peak assignments should be possible to identify the modified grease and lubricant products by analyzing corresponding witness test specimens as described earlier.

One might expect less chemical modification of the grease + lubricant additives for 385 (baseline, only 40 hr) vs. the more extensively cycled 380 and 95B specimens. However, this does not appear to be the case upon initial investigation of these surfaces. For 380, the average projected total [C] level (based on [F] and [N] marker concentrations) amounts to 34% (22% by [F], 13% by [N]), which falls well within the 38-43% range obtained for the on- and off-track surfaces.

For 95B, however, the projected total C levels, for both off- (51%) and on-track (70%) specimens exceed actual quantities detected, indicating that the additive structures have definitely been modified in favor of simpler [more stable] F- and N-rich compounds.

The FTIR spectra for specimens 380 and 95B indicated simpler, less pronounced absorbancepeaks in the normal diagnostic regions for the fluorinated grease and Schiff base lubricant materials. It is also apparent that the signal intensities for these spectra are less reduced in the "on-track" (relative to "off-track") regions of the specimens, with the weakest spectra having been generated from (on-track) specimen 95B (run to failure). This suggests a more advanced stage of degradation/combustion for surface additives inside rather than outside the wear track raceway region.

FUTURE WORK

The following recommendations are proposed to fully characterize the chemical fate of the fluorinated grease, Schiff base lubricant and bearing substrate during wear testing:

- Identify the chemical states of the additives and substrate surface by high resolution XPS at baseline (not run) and cycling (intermediate to failure) stages (see Fig. 12).

- Elemental mapping of distribution of additive markers and wear patterns on/off raceway tracks by SEM/EDS and/or SAM

- Structural *identification of organic* (grease + lubricant) compounds using IR microscope attachment of FTIR spectrometer to locate specific residual areas of interest.

- XPS/SAM sputter depth profile distribution in areas where grease is thin (depth $< 1 \mu m$) to monitor changes/effects of lubricant (and grease) on/off wear track

- Determination of film thickness vs combined roughness of rubbing surfaces using SEM/profilometry and XPS/SAM sputter depth profile techniques:

$$\text{specific film thickness} = \frac{\text{lubricant film thickness}}{\text{combined surface roughness}}$$

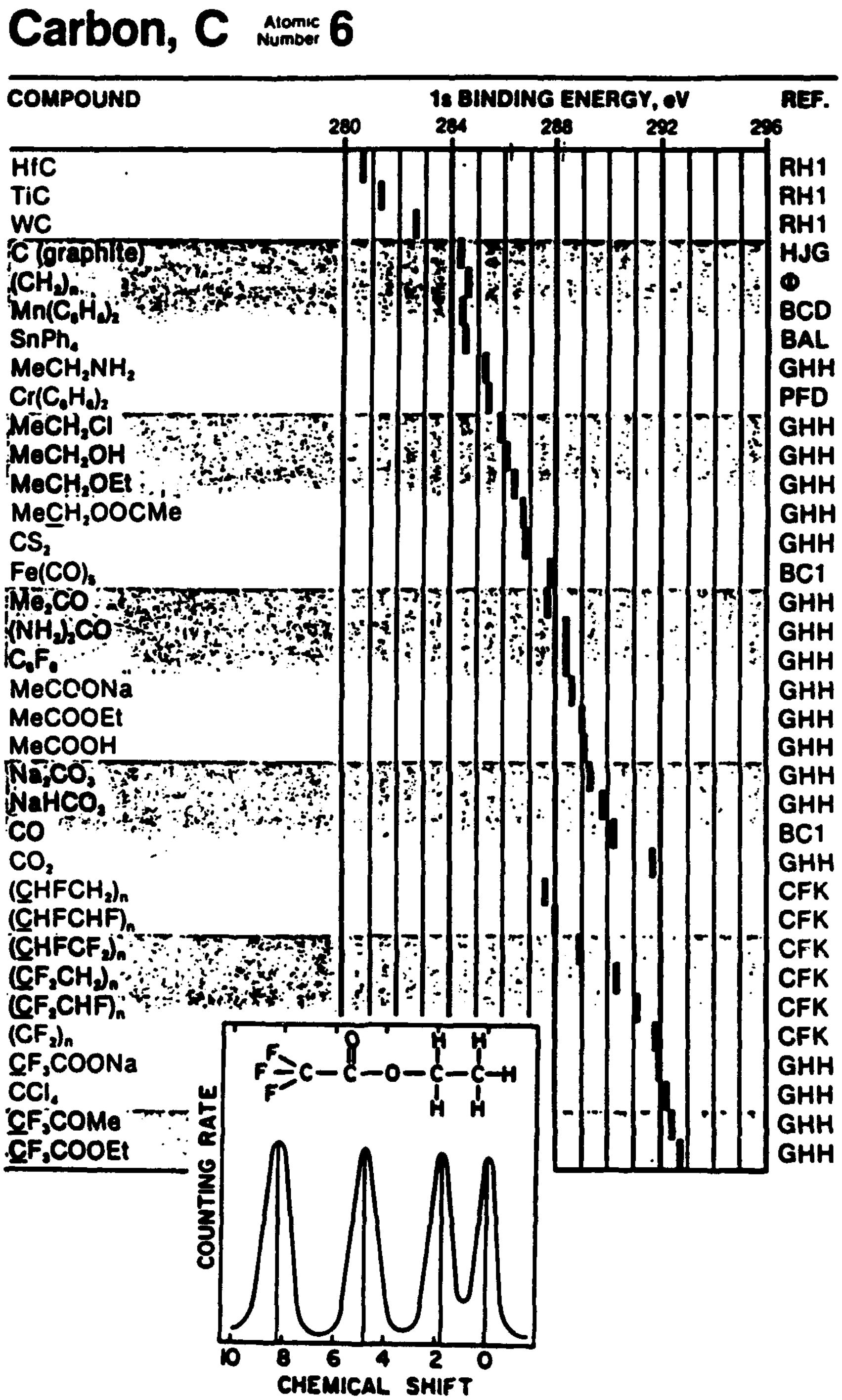


Figure 12. XPS chemical shifts for the C 1s line. The insert illustrates an example (trifluoroethylacetate) for which the four C atoms in the molecule are individually determined [Ref. C.D. Wagner, W.M. Riggs, L.E. Davis, J.F. Moulder and G.E. Muilenberg, "Handbook of X-ray Photoelectron Spectroscopy," Perkin-Elmer (1979)].

- Formulate reaction mechanism for the interaction of the Schiff base lubricant with substrate, using data from the multidisciplinary study described, to include molecular description of friction reducing and corrosion prevention action

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